

ANNEX 4

183G02 - file 2

Similar Report # 183G02
SU010398 capsule

ACCO. USA
WHEELING, ILLINOIS 60090



50505 15044 1
GREEN/VERT FEUILLE/VERDE SURVE

PHARMACIA



Analyses Report

Laboratory
Information
Management
System

Global Pharmaceutical Sciences - Italy

Submission Id: 117733

Sample ID (Request No.): 20023347

User sample ID (lot): 183G02

Sample Type: SU010398 (PNU290940AD) CAPSULE 50 MG

Request Type: STABILITY
Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 18 MONTHS

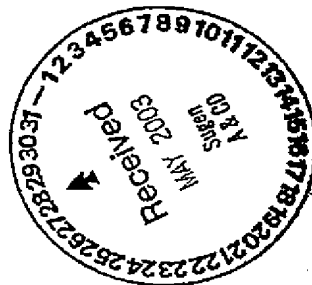
Preparation purpose: STABILITY

Condition :
APPROVED

Status :
COMPLETE

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#00	CAPSULE APPEARANCE	0=UNCHANGED	1	bd					CAPPUCCIO	MACCARI
	CAPSULE COLOR	0=UNCHANGED	1	bd					CAPPUCCIO	MACCARI
	CONTENT APPEARANCE	0=UNCHANGED	1	bd					CAPPUCCIO	MACCARI
	CONTENT COLOR	0=UNCHANGED	1	bd					CAPPUCCIO	MACCARI
	SMELL	0=UNCHANGED	1	bd					CAPPUCCIO	MACCARI
083GT01#01_S	(HPLC TITER) SAMPLE#1	100.0	1	%		90.000	110.000	15545/86-JAN 2003	CAPPUCCIO	MACCARI
	(HPLC TITER) SAMPLE#2	101.0	1	%		90.000	110.000	15545/86-JAN 2003	CAPPUCCIO	MACCARI
	(HPLC TITER) SAMPLE#3	100.6	1	%		90.000	110.000	15545/86-JAN 2003	CAPPUCCIO	MACCARI
	AVERAGE TITER (HPLC)	100.53	1	%		90.000	110.000	15545/86-JAN 2003	CAPPUCCIO	MACCARI
	TOTAL CORRELATED SUBSTANCES	0.77	1	%				15545/86-JAN 2003	CAPPUCCIO	MACCARI
083GSC01#00	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	1	%				15545/86-JAN 2003	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #1	1.9	1	%				15545/86-JAN 2003	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #2	1.8	1	%				15545/86-JAN 2003	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #3	1.8	1	%				15545/86-JAN 2003	CAPPUCCIO	MACCARI
	AVERAGE HUMIDITY	1.83	1	%				15545/86-JAN 2003	CAPPUCCIO	MACCARI



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PHARMACIA

Global Pharmaceutical Sciences - Italy

Analyses Report

Laboratory
Information
Management
System

Submission Id: 117739

Sample ID (Request No.): 20023347

User sample ID (lot): I83G02

Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG

Request Type: STABILITY

Stability Study: I83G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 18 MONTHS

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Samples Notes:

Method	Controls	Results	Vers.	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE#1	109.0	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#2	102.2	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#3	106.8	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#4	104.3	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#5	106.5	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	SAMPLE#6	105.0	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	DISSOLUTION	105.63	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	2.342	1	std-1				15545/86- JAN 2003	CAPPUCCIO	MACCARI
	C.V.	2.22	1	%				15545/86- JAN 2003	CAPPUCCIO	MACCARI
	MIN. DISSOLUTION VALUE	102.2	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI
	MAX. DISSOLUTION VALUE	109.0	1	%		80.000		15545/86- JAN 2003	CAPPUCCIO	MACCARI

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Submission Id: 117733

Sample ID (Request No.): 2002347

User sample ID (lot) : **183G02**

Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG

Request Type: STABILITY

Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 18 MONTHS

Preparation purpose: STABILITY

Condition :
APPROVED

Status :
COMPLETE

Samples Notes:

Observations on the Method 083GG02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GG01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Total correlated substances: 0.77

RRT	Area %
0.65	0.08
0.89	0.06
0.91	0.10
0.94	0.05
1.06	0.11
1.10	0.06
1.28	0.09
1.29	0.11
1.70	0.11

INTERNAL USE ONLY

SAMPLE APPROVED BY: GIUSEPPE MACCARI

APPROVE DATE: 26-may-2003

Time Points

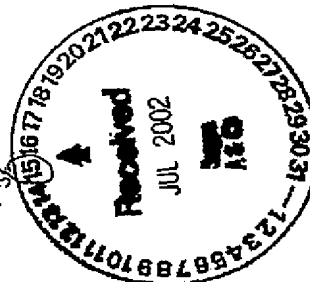
Submission Id: 116558
Sample ID (Request No.): 20021516
User sample ID (lot): **183G02**
Sample Date : 31/05/2002
Sample Type: SUJ010398 (PNU290940AD) CAPSULE 50 MG
Stability Study Start Date : 31/05/2001
S.P.N. 2001005 - Manufacturer PD/PHARMACIA Nerviano - API Batch (A)5975-MTM-0002

Batch Manufacture Date : 17/05/2001
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 12 MONTHS
Preparation purpose: STABILITY

Condition :
APPROVED
Status :
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#0	CAPSULE APPEARANCE	0=UNCHANGED	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	CAPSULE COLOR	0=UNCHANGED	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	CONTENT APPEARANCE	0=UNCHANGED	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	CONTENT COLOR	0=UNCHANGED	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SMELL	0=UNCHANGED	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
083GTI01#01	(HPLC TITER) SAMPLE#1	100.06	%		90.000	110.000	15545/27-JUN 2002	CAPPUCCIO	MACCARI
	(HPLC TITER) SAMPLE#2	99.11	%		90.000	110.000	15545/27-JUN 2002	CAPPUCCIO	MACCARI
	(HPLC TITER) SAMPLE#3	98.04	%		90.000	110.000	15545/27-JUN 2002	CAPPUCCIO	MACCARI
	AVERAGE TITER (HPLC)	99.07	%		90.000	110.000	15545/27-JUN 2002	CAPPUCCIO	MACCARI
083GSC01#0	TOTAL CORRELATED SUBSTANCES	0.80	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.20	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #2	2.25	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #3	2.25	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	AVERAGE HUMIDITY	2.23	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI



Submission Id: 116558
Sample ID (Request No.): 20021516
User sample ID (lot): I83G02
Sample Date: 31/05/2002
Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG
Stability Study Start Date: 31/05/2001
S.P.N. 2001005 - Manufacturer PDIPHARMACIA Nerviano - API Batch
(A)5975-MTM-0002

Batch Manufacture Date: 17/05/2001
Stability Study: I83G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 12 MONTHS
Preparation purpose: STABILITY

Condition: **APPROVED**
Status: **COMPLETE**

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#0 1	SAMPLE#1	92.80	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SAMPLE#2	95.91	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SAMPLE#3	96.76	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SAMPLE#4	100.16	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SAMPLE#5	99.02	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SAMPLE#6	98.74	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	DISSOLUTION	97.23	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	2.67	std-1				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	C.V.	2.75	%				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	MIN. DISSOLUTION VALUE	92.80	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
	MAX. DISSOLUTION VALUE	100.16	%		80.000		15545/27- JUN 2002	CAPPUCCIO	MACCARI
083GMC01#0 1	TOTAL AEROBIC BACTERIA	IN LIMITS	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	ESCHERICHIA COLI	ABSENT	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	SALMONELLA	ABSENT	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	FUNGI	IN LIMITS	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	STAPHYLOCOCCUS AUREUS	ABSENT	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI
	ENTEROBACTERIA	IN LIMITS	bt				15545/27- JUN 2002	CAPPUCCIO	MACCARI

Condition :
APPROVED

Status :
COMPLETE

Batch Manufacture Date : 17/05/2001

Stability Study: I83G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: STABILITY

Submission Id: 116558

Sample ID (Request No.): 20021516

User sample ID (lot): I83G02

Sample Date : 31/05/2002

Sample Type: SUJ010398 (PNUJ290940AD) CAPSULE 50 MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer: PD/PHARMACIA Naviano - API Batch

(A)S975-MTM-0002

Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored shaped hard gelatin capsule containing yellow-orange colored powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Average dissolution at 15 minutes: 96.05% (interval: 90.25%-100.16%)

Average dissolution at 45 minutes: 98.18% (interval: 91.95%-101.57%)

Observations on the Method 083GMC01#01, component: TOTAL AEROBIC BACTERIA

Total bacteria count < 10 Ufc/g (Report No. 02.9470)

Observations on the Method 083GMC01#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g (Report No. 02.9470)

Observations on the Method 083GMC01#01, component: ESCHERICHIA COLI

Report No. 02.9470

Observations on the Method 083GMC01#01, component: FUNGI

Fungi (Yeasts + Molds) < 10 Ufc/g (Report No. 02.9470)

Observations on the Method 083GMC01#01, component: SALMONELLA

Report No. 02.9470

Observations on the Method 083GMC01#01, component: STAPHYLOCOCCUS

AUREUS

Report No. 02.9470

Observations on the Method 083GCO1#00, component: IMPURITIES NOT LESS

THAN 0.05%

PHARMACIA

Pharmaceutical Development

Integrated Analytical Bulletin

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Submission Id: 116558

Sample ID (Request No.): 20021516

User sample ID (lot): I83G02

Sample Date : 31/05/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer PD/PHARMACIA Nerviano - API Batch
(A)5975-MTM-0002

Samples Notes:

Batch Manufacture Date : 17/05/2001

Stability Study: I83G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 12 MONTHS

Preparation purpose: STABILITY

Condition :
APPROVED

Status :
COMPLETE

Observations on the Method 083GC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Total correlated substances: 0.80%

RRT	Area (%)
0.65	0.10
0.90	0.05
0.91	0.09
0.94	0.05
1.00	99.20 (SU 11248)
1.06	0.14
1.09	0.06
1.28	0.07
1.29	0.11
1.70	0.12

Approved by: MACCARI
Approval date: 21-jun-2002



Pharmacia & Upjohn

Pharmaceutical Development

Analyses Report

Laboratory
Information
Management
System

Submission No: 113751
Sample ID (Request No.): 20011710
User sample ID (Lot): 183G02

Sample Type: 6U010098 (FNU20004040) CAPSULE 50 MG

Stability Study: 183G02
Pack Type: BULK
Storage Condition: TIME ZERO
Time Label: TIME ZERO
Preparation purpose: STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

Mod	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083G000110 1	APPEARANCE	COMPLIANT	bd				75/85 - JUN 2001	PASTO	MARIANI
013G02011001	HPLC IDENTIFICATION	POSITIVE	bd				75/85 - JUN 2001	PASTO	MARIANI
013G02011001 _R	(HPLC TITER) SAMPLE#1	98.3	%		95.000	105.000	75/85 - JUN 2001	PASTO	MARIANI
	(HPLC TITER) SAMPLE#2	97.7	%		95.000	105.000	75/85 - JUN 2001	PASTO	MARIANI
	(HPLC TITER) SAMPLE#3	98.1	%		95.000	105.000	75/85 - JUN 2001	PASTO	MARIANI
	AVERAGE TITER (HPLC)	98.03	%		95.000	105.000	75/85 - JUN 2001	PASTO	MARIANI
083G000110 0	TOTAL CORRELATED SUBSTANCES	1.3	%				75/85 - JUN 2001	PASTO	MARIANI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				75/85 - JUN 2001	PASTO	MARIANI
013G02011000	(HUMIDITY) SAMPLE #1	1.45	%				75/85 - JUN 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #2	1.43	%				75/85 - JUN 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #3	1.57	%				75/85 - JUN 2001	PASTO	MARIANI
	AVERAGE HUMIDITY	1.50	%				75/85 - JUN 2001	PASTO	MARIANI



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Analyses Report

Laboratory
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Subsidiary Id: 113751
Sample ID (Request No.): 20011710

User sample ID (lot): 183G02

Sample Type: SUJ10366 (FNUJ20060404) CAPSULE 50
MG

Stability Study: 183G02

Pack Type: BULK

Storage Condition: TIME ZERO

Time Label: TIME ZERO

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GUP0160 1	SAMPLE #1	97.4	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #2	101.2	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #3	101.3	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #4	100.3	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #5	102.2	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #6	96.2	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #7	96.4	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #8	97.2	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #9	96.5	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #10	105.8	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #11	97.3	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #12	97.3	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #13	96.2	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #14	103.7	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #15	99.2	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #16	96.7	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #17	95.8	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #18	99.8	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #19	96.3	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI
	SAMPLE #20	97.5	%		85.000	115.000	75/85 - JUN 2001	PASTO	MARIANI



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Analyses Report

Laboratory
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Submission Id: 113751

Sample ID (Request No.): 20011710

User sample ID (Lot): 1137502

Sample Type: SL1010368 (FNU2218404) CAPSULE 50
NG

Stability Study: 113752

Pack Type: BULK

Storage Condition: TIME ZERO

Time Label: TIME ZERO

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
063GUP0110 1	SAMPLE #20 AVERAGE WEIGHT STANDARD DEVIATION C.V. MINIMUM VALUE MAXIMUM VALUE	97.6 98.61 2.922 2.98 95.5 105.6	% % cod-1 % % %		85.000 - 115.000 86.000 - 115.000 85.000 - 115.000 85.000 - 115.000		75/85 - JUN 2001 75/85 - JUN 2001 75/85 - JUN 2001 75/85 - JUN 2001 75/85 - JUN 2001 75/85 - JUN 2001	PASTO PASTO PASTO PASTO PASTO	MARIAN MARIAN MARIAN MARIAN MARIAN MARIAN
063GDS0110 1_12									
063GMC0110 1	TOTAL AEROBIC BACTERIA ESCHERICHIA COLI SALMONELLA FUNGI STAPHYLOCOCCUS AUREUS ENTEROBACTERIA	IN LIMITS ABSENT ABSENT IN LIMITS ABSENT ABSENT	bt bt bt bt bt bt				BIOLAB MAY 2001 BIOLAB MAY 2001 BIOLAB MAY 2001 BIOLAB MAY 2001 BIOLAB MAY 2001 BIOLAB MAY 2001	PASTO PASTO PASTO PASTO PASTO PASTO	MARIAN MARIAN MARIAN MARIAN MARIAN MARIAN



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Pharmaceutical Development

Analyses Report

Laboratory
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Submission ID: 149751
Sample ID (Request No.): 20011710
User sample ID (lot): 183G02

Sample Type: SUCTION (P18120940A1) CAPSULE 50
M3

Stability Study: 183G02
Pack Type: BULK
Storage Condition: TIME ZERO
Test Label: TIME ZERO
Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
033G050140 1_12	SAMPLE #1	97.5	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #2	100.5	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #3	98.0	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #4	95.5	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #5	99.9	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #6	97.2	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #7	98.7	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #8	99.3	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #9	97.8	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #10	98.7	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #11	99.0	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	SAMPLE #12	99.0	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	DISSOLUTION	98.51	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	STANDARD DEVIATION	1.325	std-1				75/95 - JUN 2001	PASTO	MARIANI
	C.V.	1.36	%				75/95 - JUN 2001	PASTO	MARIANI
	MIN. DISSOLUTION VALUE	95.5	%		80.000		75/95 - JUN 2001	PASTO	MARIANI
	MAX. DISSOLUTION VALUE	100.5	%		80.000		75/95 - JUN 2001	PASTO	MARIANI



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Analyses Report

Laboratory
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Submission ID: 113751

Sample ID (Request No.): 20011710

User sample ID (lot): 183302

Sample Type: SU010398 (N12800-00AD) CAPSULE 50 MG

Stability Study: 183302

Pack Type: BULK

Storage Condition: TIME ZERO

Time Label: TIME ZERO

Preparation purpose: STABILITY

Condition:

APPROVED

Status:

COMPLETE

Samples Notes:

Observations on the Method 083GCO1#01, component: APPEARANCE

Opaque brick red colored shaped hard gelatin capsule containing yellow-orange colored powder

Observations on the Method 083GDS01#01_12, component: MAXIMUM DISSOLUTION VALUE

no comment

Observations on the Method 083GDS01#01_12, component: DISSOLUTION

Average dissolution at 15 minutes: 97.7% (C.V. 2.6%)

Average dissolution at 30 minutes: 96.5% (C.V. 1.4%)

Average dissolution at 45 minutes: 99.5% (C.V. 1.7%)

Observations on the Method 083GMC01#01, component: TOTAL AEROBIC BACTERIA

Total bacteria count < 10 Ufc/g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: ESCHERICHIA COLI

Test done on 10g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: FUNGI

Fungi (yeasts and molds) < 10 Ufc/g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: SALMONELLA

Test done on 10g (Report No. 01.1231)

Observations on the Method 083GMC01#01, component: STAPHYLOCOCCUS AUREUS

Report No. 01.1231

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

correlated substances
RRT 0.690 (impurities A): 0.16%

Printed 13-JUN-2001 01:50 PM by MAEYAN



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Analyses Report

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Submission Id: 113751

Sample ID (Request No.) : 20011710

User sample ID (lot) : 199G02

Sample Type: SUB0398 (PNU2009-04AD) CAPSULE 50
MG

Stability Study: 183902

Pack Type: BULK

Storage Condition: TIME ZERO

Time Label: TIME ZERO

Preparation purpose: STABILITY

Condition:

APPROVED

Status:

COMPLETE

Samples Notes:

Observations on the Method 083GC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

RRT 0.913 (IMPURITIES B): 0.08 %
RRT 0.925 (IMPURITIES C): 0.15 %
RRT 1.059 (IMPURITIES D): 0.25 %
RRT 1.069 (IMPURITIES E): 0.11 %
RRT 1.142 (IMPURITIES F): 0.07 %
RRT 1.281 (IMPURITIES G): 0.13 %
RRT 1.272 (IMPURITIES H): 0.15 %
RRT 1.646 (IMPURITIES I): 0.16 %

Observations on the Method 083GUP01#01, component: AVERAGE WEIGHT
The average weight is equal to 98.61% which corresponds to 87.85 mg per capsule.

Approved by: MARIANI
Approval date: 13-Jun-2001

Integrated Analytical Bulletin

Pharmacia & Upjohn

Pharmaceutical Development

Condition: **APPROVED**
Status: **COMPLETE**

Batch Manufacture Date: 17/05/2001
Stability Study: 189302
Pack Type: 30 ml PLASTIC BOTTLE
Storage Conditions: 25°C/60% U.R.
Time Label: 1 MONTH
Preparation purpose: STABILITY

Submission Id: 113892
Sample ID (Request No.): 20072018
User sample ID (lot): 203G02
Sample Date: 30/08/2001
Sample Type: SU010308 (PAU220840AD) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001
S.P.N. 2007DVS - Manufacturer PDFTAPMACIA Nórdico - API Batch (A5675-MRUP-0002)

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
063G0002M0	CAPSULE APPEARANCE	0=UNCHANGED	bd				1544101 - AUG 2001	COLOMBOD	MARIAN
	CAPSULE COLOR	0=UNCHANGED	bd				1544101 - AUG 2001	COLOMBOD	MARIAN
	CONTENT APPEARANCE	0=UNCHANGED	bd				1544101 - AUG 2001	COLOMBOD	MARIAN
	CONTENT COLOR	0=UNCHANGED	bd				1544101 - AUG 2001	COLOMBOD	MARIAN
	SMELL	0=UNCHANGED	bd				1544101 - AUG 2001	COLOMBOD	MARIAN
063G0104M1	(HPLC TITER) SAMPLE#1	98.4	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIAN
	(HPLC TITER) SAMPLE#2	98.1	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIAN
	(HPLC TITER) SAMPLE#3	98.0	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIAN
	AVERAGE TITER (HPLC)	98.17	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIAN
063G0301M0	TOTAL CORRELATED SUBSTANCES	1.04	%				1544103 - AUG 2001	COLOMBOD	MARIAN
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				1544103 - AUG 2001	COLOMBOD	MARIAN
063G0401M0	(HUMIDITY) SAMPLE #1	2.0	%				1544103 - AUG 2001	COLOMBOD	MARIAN
	(HUMIDITY) SAMPLE #2	2.0	%				1544103 - AUG 2001	COLOMBOD	MARIAN
	(HUMIDITY) SAMPLE #3	2.0	%				1544103 - AUG 2001	COLOMBOD	MARIAN
	AVERAGE HUMIDITY	2	%				1544103 - AUG 2001	COLOMBOD	MARIAN



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Submission Id: 113002

Sample ID (Request No.): 20012040

User sample ID (lot): 103G02

Sample Date: 30/08/2001

Sample Type: S1010398 (7410220840A0) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001

S.P.N. 2001005 - Manufacture POPHARMACIA Nerviano - API Batch (A)975-MTM-0002

Samples Notes:

Batch: Manufacture Date: 17/05/2001

Stability Study: 103G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% RH.

Time Label: 1 MONTH

Preparation purpose: STABILITY

Condition: **APPROVED**

Status: **COMPLETE**

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
003GDS0110 1	SAMPLE #1	101.4	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	SAMPLE #2	103.2	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	SAMPLE #3	100.8	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	SAMPLE #4	98.7	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	SAMPLE #5	98.7	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	SAMPLE #6	97.9	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	DISSOLUTION	100.87	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	STANDARD DEVIATION	1.789	std-1				15441/4 SEP 2001	PASTO	MARIANI
	C.V.	1.78	%				15441/4 SEP 2001	PASTO	MARIANI
	MIN. DISSOLUTION VALUE	97.9	%		80.000		15441/4 SEP 2001	PASTO	MARIANI
	MAX. DISSOLUTION VALUE	103.2	%		80.000		15441/4 SEP 2001	PASTO	MARIANI



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Submission ID: 113602

Sample ID (Request No.): 20012049

User sample ID (lot): 083G02

Sample Date: 2000/05/2001

Sample Type: SU010203 (PML230040AD) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001

S.P.N. 2001005 - Manufacturer PDHARMACIA Nordinco - API Batch
A5975MTM-0002

Samples Notes:

Condition:
APPROVED

Status:
COMPLETE

Batch Manufacture Date: 17/05/2001

Stability Study: 083G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 1 MONTH

Preparation purpose: STABILITY

Observations on the Method 083GDS01#01, component: DISSOLUTION
average dissolution per single capsule after 15 minutes: 96.5% (C.V. 2.0%)
average dissolution per single capsule after 30 minutes: 100.4% (C.V. 1.8%)
average dissolution per single capsule after 45 minutes: 100.7% (C.V. 2.0%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

CORRELATED SUBSTANCES

RRT 0.967 : 0.05%
RRT 0.915 : 0.05%
RRT 0.938 : 0.11%
RRT 1.054 : 0.23%
RRT 1.064 : 0.06%
RRT 1.257 : 0.06%
RRT 1.267 : 0.18%
RRT 1.845 : 0.13%

Approved by: MARRANI
Approval date: 07-sep-2001



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Submission Id: 113814

Sample ID (Request No.): 2012351

User sample ID (lot): 183502

Sample Date: 30/08/2001

Sample Type: 81010368 (PNU2908 404D) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001

S.P.N. 20101005 - Manufacture PDPHARMACIA Nordano - API Batch (A9975-MTAT-0002)

Batch Manufacture Date: 17/05/2001

Stability Study: 183502

Pack Type: 30 ml PLASTIC BOTTLE

Storage Conditions: 40°C/75% U.R.

Time Label: 1 MONTH

Preparation purpose: STABILITY

Condition: **APPROVED**

Status: **COMPLETE**

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data	Analyses	Analyst	Verifier
083G000240 0	CAPSULE APPEARANCE	0=UNCHANGED	td				1544102 - AUG 2001	COLOMBOD	MARIANI	
	CAPSULE COLOR	0=UNCHANGED	td				1544102 - AUG 2001	COLOMBOD	MARIANI	
	CONTENT APPEARANCE	0=UNCHANGED	td				1544102 - AUG 2001	COLOMBOD	MARIANI	
	CONTENT COLOR	0=UNCHANGED	td				1544102 - AUG 2001	COLOMBOD	MARIANI	
	SMELL	0=UNCHANGED	td				1544102 - AUG 2001	COLOMBOD	MARIANI	
083G1101401 S	(HPLC TITER) SAMPLE#1	97.9	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIANI	
	(HPLC TITER) SAMPLE#2	95.7	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIANI	
	(HPLC TITER) SAMPLE#3	98.1	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIANI	
	AVERAGE TITER (HPLC)	97.57	%		90.000	110.000	1544102 - AUG 2001	COLOMBOD	MARIANI	
083G5C0140 0	TOTAL CORRELATED SUBSTANCES	1.21	%				1544103 - AUG 2001	COLOMBOD	MARIANI	
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				1544103 - AUG 2001	COLOMBOD	MARIANI	
083G6F0140C	(HUMIDITY) SAMPLE #1	1.9	%				1544103 - AUG 2001	COLOMBOD	MARIANI	
	(HUMIDITY) SAMPLE #2	2.1	%				1544103 - AUG 2001	COLOMBOD	MARIANI	
	(HUMIDITY) SAMPLE #3	2.0	%				1544103 - AUG 2001	COLOMBOD	MARIANI	
	AVERAGE HUMIDITY	2	%				1544103 - AUG 2001	COLOMBOD	MARIANI	



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Submission Ref: 113994

Sample ID (Request No.): 20012051

User sample ID (lot): 103G02

Sample Date: 30/06/2001

Sample Type: SLA10398 (PML29/09/00AD) CAPSULE 50
MG

Stability Study Start Date: 31/05/2001

S.P.N.: 2001005 - Manufacturer PD/PHARMACIA Nerviano - API Batch
(A9375-MTU-0012)

Samples Notes:

Batch Manufacture Date: 17/05/2001

Stability Study: 103902

Pack Type: 30 ml PLASTIC BOTTLE

Storage Conditions: 40°C/75% U.R.

Time Label: 1 MONTH

Preparation purpose: STABILITY

Condition:

APPROVED

Status:

COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
03362501#0 1	SAMPLE #1	98.9	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	SAMPLE #2	95.7	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	SAMPLE #3	98.0	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	SAMPLE #4	96.9	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	SAMPLE #5	95.2	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	SAMPLE #6	97.5	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	DISSOLUTION	97.2	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	STANDARD DEVIATION	1.655	std-1				1544/14 SEP 2001	PASTO	MARIANI
	C.V.	1.63	%				1544/14 SEP 2001	PASTO	MARIANI
	MIN. DISSOLUTION VALUE	95.2	%		80.000		1544/14 SEP 2001	PASTO	MARIANI
	MAX. DISSOLUTION VALUE	99.0	%		80.000		1544/14 SEP 2001	PASTO	MARIANI



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Submission ID: 113994

Sample ID (Request No.): 20012051

User sample ID (lot): 183G02

Sample Date: 30/05/2001

Sample Type: S1J010358 (PNU28014-0AD) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001

S.P. AL 2801005 - Manufacturer PDFF-HARMACIA Verlamu - API Batch (A)875-MITH-0002

Samples Notes:

Batch Manufacture Date: 17/05/2001

Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% D.R.

Time Lapse: 1 MONTH

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Observations on the Method 083GDS01#01, component: DISSOLUTION

average dissolution per single capsule after 15 minutes: 96.0% (CV 1.5%)

average dissolution per single capsule after 30 minutes: 97.2% (CV 1.6%)

average dissolution per single capsule after 45 minutes: 97.3% (CV 1.6%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS

THAN 0.05%

CORRELATED SUBSTANCES

RRT 0.891: 0.03%

RRT 0.916: 0.00%

RRT 0.928: 0.13%

RRT 0.956: 0.05%

RRT 1.064: 0.34%

RRT 1.083: 0.16%

RRT 1.260: 0.08%

RRT 1.270: 0.15%

RRT 1.655: 0.14%

Approved by: **MARIANI**
Approval date: 07-sep-2001

Submission Id: 115782
Sample ID (Request No.): 20020525
User sample ID (lot): 183G02

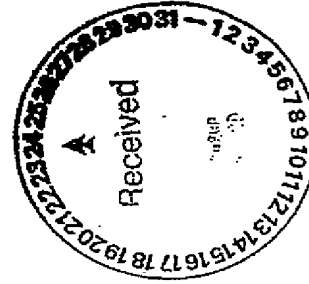
Sample Type: SUJ0398 (PNU280940AD) CAPSULE 50
MG

Request Type: RELEASE
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 9 MONTHS
Preparation purpose: STABILITY

Condition:
APPROVED
Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCO02#0	CAPSULE APPEARANCE	CONFORMS	td				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	CAPSULE COLOR	CONFORMS	td				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	CONTENT APPEARANCE	CONFORMS	td				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	CONTENT COLOR	CONFORMS	td				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	SMELL	CONFORMS	td				1554/08- MAR 2002	CAPPUCCIO	MARIANI
083GTI01#01	(HPLC TITER) SAMPLE#1	100.2	%		90.000	110.000	1554/08- MAR 2002	CAPPUCCIO	MARIANI
S	(HPLC TITER) SAMPLE#2	99.5	%		90.000	110.000	1554/08- MAR 2002	CAPPUCCIO	MARIANI
	(HPLC TITER) SAMPLE#3	99.4	%		90.000	110.000	1554/08- MAR 2002	CAPPUCCIO	MARIANI
	AVERAGE TITER (HPLC)	99.7	%		90.000	110.000	1554/08- MAR 2002	CAPPUCCIO	MARIANI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	0.81	%				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				1554/08- MAR 2002	CAPPUCCIO	MARIANI
083GKF01#00	(HUMIDITY) SAMPLE #1	2.2	%				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	(HUMIDITY) SAMPLE #2	2.1	%				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	(HUMIDITY) SAMPLE #3	2.2	%				1554/08- MAR 2002	CAPPUCCIO	MARIANI
	AVERAGE HUMIDITY	2.17	%				1554/08- MAR 2002	CAPPUCCIO	MARIANI



Submission Id: 115782
Sample ID (Request No.): 20020525
User sample ID (lot): 183G02

Sample Type: SUJ10398 (PNU290940AD) CAPSULE 50
MG

Request Type: RELEASE
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 9 MONTHS
Preparation purpose: STABILITY

Condition:
APPROVED
Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE #1	97.15	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	SAMPLE #2	98.05	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	SAMPLE #3	99.81	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	SAMPLE #4	100.49	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	SAMPLE #5	99.57	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	SAMPLE #6	100.12	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	DISSOLUTION	99.17	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	STANDARD DEVIATION	1.291	std-1				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	C.V.	1.3	%				15545/08- MAR 2002	CAPPUCCIO	MARIANI
	MIN. DISSOLUTION VALUE	97.15	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI
	MAX. DISSOLUTION VALUE	100.49	%		80.000		15545/08- MAR 2002	CAPPUCCIO	MARIANI

Submission Id: 115762

Sample ID (Request No.) : 20020525

User sample ID (lot) : 183G02

Sample Type: SU010598 (PNU290840AD) CAPSULE 50
MG

Request Type: RELEASE

Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 9 MONTHS

Preparation purpose: STABILITY

Condition :

APPROVED

Status :

COMPLETE

Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

The dissolution values were corrected based on the weight of the respective capsule

Average dissolution at 15 minutes: (interval 96.4% - 100.1%)

Average dissolution at 45 minutes: (interval 96.6% - 100.2%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS

THAN 0.05%

Total correlated substances: 0.81%

RRT	Area %
0.65	0.08
0.89	0.06
0.91	0.12
0.94	0.05
1.00	99.19 (SU 11248)
1.06	0.20
1.10	0.10
1.28	0.07
1.30	0.13

Approved by: MARIANI
Approval date: 25-mar-2002



Pharmacia & Upjohn

Pharmaceutical Development

Integrated Analytical Bulletin

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Publication ID: 115149

Sample ID (Request No.): 20013447

User sample ID (Lot): 185G02

Sample Date: 30/11/2001

Sample Type: SU010898 (FNU23090400) CAPSULE 50 MG

Stability Study Start Date: 31/05/2001

S.P.A. 20011005 - Manufacturer FOFH/AFMACIA Novartis - API Batch (A95975-MT14-0002)

Batch Manufacture Date: 17/05/2001

Stability Study: 185G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Conditions: 25°C/60% U.L.

Time Label: 6 MONTHS

Preparation purpose: STABILITY

Condition: **APPROVED**

Status: **COMPLETE**

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083G000200	CAPSULE APPEARANCE	CONFORMS	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	CAPSULE COLOR	CONFORMS	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	CONTENT APPEARANCE	CONFORMS	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	CONTENT COLOR	CONFORMS	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	SMELL	CONFORMS	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
083G101401	(HPLC TITER) SAMPLE#1	96.57	%		90.000	110.000	14/27 DEC 2001	CAPPUCCIO	MACCARI
-S	(HPLC TITER) SAMPLE#2	96.90	%		90.000	110.000	14/27 DEC 2001	CAPPUCCIO	MACCARI
	(HPLC TITER) SAMPLE#3	98.85	%		90.000	110.000	14/27 DEC 2001	CAPPUCCIO	MACCARI
	AVERAGE TITER (HPLC)	96.44	%		90.000	110.000	14/27 DEC 2001	CAPPUCCIO	MACCARI
083G500140	TOTAL CORRELATED SUBSTANCES	0.9	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
0	IMPURITIES NOT LESS THAN 0.05%	SEE TEXT	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
083G4001400	(HUMIDITY) SAMPLE #1	1.7	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #2	1.7	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	(HUMIDITY) SAMPLE #3	1.3	%				14/27 DEC 2001	CAPPUCCIO	MACCARI
	AVERAGE HUMIDITY	1.73	%				14/27 DEC 2001	CAPPUCCIO	MACCARI



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Submission ID: 115149

Sample ID (Request No.): 20013447

User sample ID (lot) 80302

Sample Date: 30/11/2001

Sample Type: 80010398 (PNU2004040) CAPSULE 50
mg

Stability Study Start Date: 31/05/2001

S.P.N. 2001005 - Manufacturer: PO PHARMACIA Norwina - API Batch
(A)975-401N-0002

Batch Number/Date: 17/05/2001

Stability Study: 80302

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Test Label: 6 MONTHS

Preparation purpose: STABILITY

Condition:
APPROVED

Status:
COMPLETE

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data	Analyst	Verifier
083GDS0190 1	SAMPLE #1	97.9	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #2	100.2	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #3	97.4	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #4	98.8	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #5	102.7	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #6	100.2	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	DISSOLUTION	98.53	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	1.933	std-1				14927 DEC 2001	CAPPUCCIO	MACCARI
	C.V.	1.94	%				14927 DEC 2001	CAPPUCCIO	MACCARI
	MIN. DISSOLUTION VALUE	97.36	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI
	MAX. DISSOLUTION VALUE	102.7	%		80.000		14927 DEC 2001	CAPPUCCIO	MACCARI

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Condition :
APPROVED
Status :
COMPLETE

Batch Manufacture Date : 17/05/2001
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Conditions: 25°C/60% U.R.
Time Label: 8 MONTHS
Preparation purpose: STABILITY

Submission Id: 116149
Sample ID (Request No.) : 20013447
User sample ID (lot) : 183G02
Sample Date : 30/11/2001
Sample Type: SU010308 (FNU230940AD) CAPSULE 50 MG

Stability Study Start Date : 31/05/2001
S.P.N. 20010015 - Manufacturer: FORTAPHARMACIA Nordano - API Solut
(AN575-MTN-0002)

Samples Notes:

Observations on the Method 083GCO2#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.9%

RRT	Area %
0.90	0.006
0.90	0.002
0.91	0.110
1.00	98.006
1.05	0.124
1.27	0.138
1.28	0.110
1.69	0.173
2.17	0.134

Approved by: MACCARI
Approval date: 11-dec-2001

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Pharmaceutical Development

Condition :
APPROVED
Status :
COMPLETE

Batch Manufacture Date : 17/05/2001
Stability Study: ISS002
Pack Type: 30 ml PLASTIC BOTTLE
Storage Conditions: 40°C/75% U.R.
Time Label: 6 MONTHS
Preparation purpose: STABILITY

Submission Id: 115151
Sample ID (Request No.) : 20013449
User sample ID (lot) : 183G02
Sample Date : 30/11/2001
Sample Type: SU010398 (PNU200940AD) CAPSULE 50 MG

Stability Study Start Date : 31/05/2001
S.P.N. 20014005 - Manufacture PDFFARMACIA Navarino - API Batch
(A)9975-R11M-0002

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
0636000240	CAPSULE APPEARANCE	CONFORMS	bt				14/27 DEC 2001	CAPPUCIO	MACCARI
0	CAPSULE COLOR	CONFORMS	bt				14/27 DEC 2001	CAPPUCIO	MACCARI
	CONTENT APPEARANCE	CONFORMS	bt				14/27 DEC 2001	CAPPUCIO	MACCARI
	CONTENT COLOR	CONFORMS	bt				14/27 DEC 2001	CAPPUCIO	MACCARI
	SMELL	CONFORMS	bt				14/27 DEC 2001	CAPPUCIO	MACCARI
0636000140	(HPLC TITER) SAMPLE#1	98.57	%		90.000	110.000	14/27 DEC 2001	CAPPUCIO	MACCARI
-5	(HPLC TITER) SAMPLE#2	98.57	%		90.000	110.000	14/27 DEC 2001	CAPPUCIO	MACCARI
	(HPLC TITER) SAMPLE#3	100.00	%		90.000	110.000	14/27 DEC 2001	CAPPUCIO	MACCARI
	AVERAGE TITER (HPLC)	99.41	%		90.000	110.000	14/27 DEC 2001	CAPPUCIO	MACCARI
0636000140	TOTAL CORRELATED SUBSTANCES	0.57	%				14/27 DEC 2001	CAPPUCIO	MACCARI
0	IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%				14/27 DEC 2001	CAPPUCIO	MACCARI
0636000140	(HUMIDITY) SAMPLE #1	2.2	%				14/27 DEC 2001	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #2	2.2	%				14/27 DEC 2001	CAPPUCIO	MACCARI
	(HUMIDITY) SAMPLE #3	2.2	%				14/27 DEC 2001	CAPPUCIO	MACCARI
	AVERAGE HUMIDITY	2.2	%				14/27 DEC 2001	CAPPUCIO	MACCARI

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Submission ID: 115151
Sample ID (Request No.): 20013440
User sample ID (lot): 113302
Sample Date: 30/11/2001
Sample Type: SUB010398 (FNU25064040) CAP8JULE 50 MG

Batch Manufacture Date: 17/03/2001
Stability Study: 85302

Pack Type: 30 ml PLASTIC BOTTLE
Storage Conditions: 40°C/75% U.R.
Time Label: 8 MONTHS

Preparation purpose: STABILITY

Stability Study Start Date: 31/03/2001

S.P.N. 2001005 - Manufacture PDFTAPBULACIA Números - API Batch
1A3575-MTM-0002

Samples Notes:

Condition:
APPROVED

Status:
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
0598D50140	SAMPLE #1	83.2	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #2	102.2	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #3	101.9	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #4	101.8	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #5	102.7	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	SAMPLE #6	99.9	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	DISSOLUTION	101.08	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	STANDARD DEVIATION	1.703	std-1				14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	C.V.	1.68	%				14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	MIN. DISSOLUTION VALUE	98.2	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI
	MAX. DISSOLUTION VALUE	102.7	%		80.000		14/3/27 DEC 2001	CAPPUCCIO	MACCARI

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Condition: **APPROVED**
Status: **COMPLETE**

Batch Manufacture Date: 17/05/2001
Stability Study: 183602
Pack Type: 30 ml PLASTIC BOTTLE
Storage Conditions: 40°C/75% UR.
Time Label: 6 MONTHS
Preparation purpose: STABILITY

Submission ID: 115151
Sample ID (Request No.): 20018449
User sample ID (lot): 183602
Sample Date: 30/11/2001
Sample Type: 84310808 (PMU20004WD) CAPSULE 60
MG

Stability Study Start Date: 31/05/2001
S.P.N. 2001006 - Manufacturer PDH/HARMACIA Novartis - API Batch
(A567C-MTN-0002)

Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Total correlated substances: 0.97%

IMP	Area %
0.86	0.06
0.90	0.05
0.91	0.13
1.00	99.03
1.05	0.19
1.08	0.05
1.27	0.14
1.28	0.11
1.89	0.12
2.17	0.12

Approved by: MACCARI
Approval date: 11-dec-2001



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Submission ID: 114395
Sample ID (Request No.): 20012535
User sample ID (lot): 183G02
Sample Date: 31/08/2001
Sample Type: SU010388 (PNU280940AD) CAPSULE 50
MG

Batch Manufacture Date: 17/05/2001
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Label: 3 MONTHS
Preparation purpose: STABILITY

Condition: APPROVED
Status: COMPLETE

Stability Study Start Date: 31/05/2001
S.P.N. 2001005 - Manufacturer PD/PHARMACIA Nerviano - API Batch
(A)5575-MTM-0002

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GSC02100	CAPSULE APPEARANCE	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
0	CAPSULE COLOR	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	CONTENT COLOR	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	SMELL	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
083GT01001	(HPLC TITER) SAMPLE#1	99.1	%		80.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
S	(HPLC TITER) SAMPLE#2	100.1	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
	(HPLC TITER) SAMPLE#3	100.6	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
	AVERAGE TITER (HPLC)	99.93	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
083GSC01000	TOTAL CORRELATED SUBSTANCES	0.96	%				15441/27 SEP 2001	PASTO	MARIANI
	IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%				15441/27 SEP 2001	PASTO	MARIANI
083GKF01000	(HUMIDITY) SAMPLE #1	1.5	%				15441/27 SEP 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #2	1.9	%				15441/27 SEP 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #3	1.4	%				15441/27 SEP 2001	PASTO	MARIANI
	AVERAGE HUMIDITY	1.6	%				15441/27 SEP 2001	PASTO	MARIANI



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Submission Id: 114395

Sample ID (Request No.): 20012536

User sample ID (lot): IB3G02

Sample Date : 31/08/2001

Sample Type: SU010398 (PNU280940AD) CAPSULE 50
MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer: PD/PHARMACIA Naviano - API Batch

(A)5875-MTM-0002

Samples Notes:

Batch Manufacture Date : 17/05/2001

Stability Study: IB3G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 3 MONTHS

Preparation purpose: STABILITY

Condition :
APPROVED

Status :
COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01W01	SAMPLE #1	98.6	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	SAMPLE #2	99.1	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	SAMPLE #3	98.0	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	SAMPLE #4	98.9	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	SAMPLE #5	99.5	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	SAMPLE #6	97.8	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	DISSOLUTION	97.32	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	STANDARD DEVIATION	1.713	std-1				15441/31 SEP 2001	PASTO	MARIANI
	C.V.	1.76	%				15441/31 SEP 2001	PASTO	MARIANI
	MIN. DISSOLUTION VALUE	95.1	%		80.000		15441/31 SEP 2001	PASTO	MARIANI
	MAX. DISSOLUTION VALUE	98.5	%		80.000		15441/31 SEP 2001	PASTO	MARIANI



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Submission Id: 114395

Sample ID (Request No.): 20012535

User sample ID (lot): 183G02

Sample Date: 31/08/2001

Sample Type: SU010398 (PNU280940AD) CAPSULE 50
MG

Stability Study Start Date: 31/05/2001

S.P.N. 2001005 - Manufacturer PD/PHARMACIA Nerviano - API Batch

(A)575-MTM-0002

Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

average dissolution per single capsule after 15 minutes: 96.4% (C.V. 1.5%)

average dissolution per single capsule after 30 minutes: 96.4% (C.V. 1.8%)

average dissolution per single capsule after 45 minutes: 98.6% (C.V. 1.6%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Correlated substances

RRT 0.693 : 0.10%

RRT 0.915 : 0.05%

RRT 0.927 : 0.13%

RRT 1.059 : 0.17%

RRT 1.092 : 0.08%

RRT 1.254 : 0.09%

RRT 1.265 : 0.20%

RRT 1.635 : 0.14%

Condition: APPROVED

Status: COMPLETE

Batch Manufacture Date: 17/05/2001

Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 25°C/60% U.R.

Time Label: 3 MONTHS

Preparation purpose: STABILITY

Approved by: MARIANI

Approval date: 01-oct-2001



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Submission Id: 114397
Sample ID (Request No.): 20012537
User sample ID (lot): 183G02
Sample Date: 31/08/2001
Sample Type: SU010388 (PNU290840AD) CAPSULE 50
MG

Batch Manufacture Date: 17/06/2001
Stability Study: 183G02
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40°C/75% U.R.
Time Label: 3 MONTHS
Preparation purpose: STABILITY

Condition:
APPROVED
Status:
COMPLETE

Stability Study Start Date: 31/08/2001

S.P.N. 2001005 - Manufacturer PD/PHARMACIA Narvango - API Batch
(A)5975-MTM-0002

Samples Notes:

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GCD02#0	CAPSULE APPEARANCE	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	CAPSULE COLOR	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	CONTENT APPEARANCE	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	CONTENT COLOR	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
	SMELL	0=UNCHANGED	bt				15441/27 SEP 2001	PASTO	MARIANI
083GT01#01 S	(HPLC TITER) SAMPLE#1	99.6	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
	(HPLC TITER) SAMPLE#2	99.5	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
	(HPLC TITER) SAMPLE#3	99.2	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
	AVERAGE TITER (HPLC)	99.43	%		90.000	110.000	15441/27 SEP 2001	PASTO	MARIANI
083GSC01#00	TOTAL CORRELATED SUBSTANCES	1.07	%				15441/27 SEP 2001	PASTO	MARIANI
	IMPURITIES NOT LESS THAN 0.05% SEE TEXT		%				15441/27 SEP 2001	PASTO	MARIANI
083GKFD1#00	(HUMIDITY) SAMPLE #1	1.9	%				15441/27 SEP 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #2	2.1	%				15441/27 SEP 2001	PASTO	MARIANI
	(HUMIDITY) SAMPLE #3	2.0	%				15441/27 SEP 2001	PASTO	MARIANI
	AVERAGE HUMIDITY	2	%				15441/27 SEP 2001	PASTO	MARIANI



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Submission Id: 114397

Sample ID (Request No.): 20012537

User sample ID (lot): 183G02

Sample Date: 31/08/2001

Sample Type: SU010398 (PNU290940AD) CAPSULE 50 MG

Stability Study Start Date: 31/08/2001

S.P.N. 2001006 - Manufacturer PDPHARMACIA Nordino - API Batch

(A)5975-NITM-0002

Samples Notes:

Batch Manufacture Date: 17/05/2001

Stability Study: 183G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 3 MONTHS

Preparation purpose: STABILITY

Condition: APPROVED

Status: COMPLETE

Method	Controls	Results	M.U.	Spec?	Lower lim	Upper lim	Book-Data Analyses	Analyst	Verifier
083GDS01#01	SAMPLE #1	97.7	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	SAMPLE #2	94.5	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	SAMPLE #3	98.1	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	SAMPLE #4	95.1	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	SAMPLE #5	93.1	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	SAMPLE #6	100.1	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	DISSOLUTION	96.43	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	STANDARD DEVIATION	2.625	std-1				1544/1/31 SEP 2001	PASTO	MARIANI
	C.V.	2.72	%				1544/1/31 SEP 2001	PASTO	MARIANI
	MIN. DISSOLUTION VALUE	94.5	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI
	MAX. DISSOLUTION VALUE	100.1	%		80.000		1544/1/31 SEP 2001	PASTO	MARIANI



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Submission Id: 114397

Sample ID (Request No.) : 20012537

User sample ID (lot) : I83G02

Sample Date : 31/08/2001

Sample Type: SU010398 (PNU290940AD) CAPSULE 50
MG

Stability Study Start Date : 31/05/2001

S.P.N. 2001005 - Manufacturer PD/PHARMACIA Naviano - API Batch

(A)5975-WTM-0002

Samples Notes:

Observations on the Method

083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

average dissolution single determination after 15 minutes: 95.6% (C.V. 2.7%)

average dissolution single determination after 30 minutes: 96.4% (C.V. 2.7%)

average dissolution single determination after 45 minutes: 96.4% (C.V. 2.7%)

Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS
THAN 0.05%

Correlated substances

RRT 0.685 : 0.11%

RRT 0.915 : 0.07%

RRT 0.925 : 0.14%

RRT 0.858 : 0.07%

RRT 1.058 : 0.16%

RRT 1.080 : 0.08%

RRT 1.260 : 0.08%

RRT 1.282 : 0.21%

RRT 1.632 : 0.14%

Batch Manufacture Date : 17/05/2001

Stability Study: I83G02

Pack Type: 30 ml PLASTIC BOTTLE

Storage Condition: 40°C/75% U.R.

Time Label: 3 MONTHS

Preparation purpose: STABILITY

Condition :
APPROVED

Status :
COMPLETE

Approved by: MARIANI

Approval date: 01-oct-2001